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This paper is dedicated to the memory of Lívia Rudnyánszky.

A LABORATORY PROCEDURE FOR SEPARATING MICAS FROM QUARTZ IN CLAY-SIZED MATERIALS

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ABSTRACT

In this paper, a new laboratory procedure is presented for the separation of micas from quartz in clay-sized materials. The key part of the procedure consists of centrifuging the clay-sized material in a non-reactive, high-viscosity medium (2% solution of sodium carboxymethyl cellulose). As a result of the centrifugation, platy clay minerals stay in suspension while quartz grains settle down. After centrifugation, the water-soluble carboxymethyl cellulose can be easily washed out of the clay mineral separate.

The procedure was tested on the mixed clay-sized material of the Úrkút manganese carbonate ore (Weiszburg et al., 2004), with the phyllosilicate-quartz separation being the last step in a three-step separation process. The separation was monitored by X-ray powder diffraction, infrared spectroscopy and full chemical analysis, justifying that the separation medium is really non-reactive and celadonite, the colour-giving green mica of the Úrkút manganese carbonate ore remained practically unchanged.

The possible effect of quartz impurities on the chemical formula (calculated from full chemical analysis) of micas, and consequently, on the classification of micas is also tested on the Úrkút celadonite.

Key words: separation, mica, quartz, sodium carboxymethyl cellulose, clay-sized materials

INTRODUCTION

The preparation of pure phyllosilicate separates from mixed clay-sized materials is often problematic. Carbonates and goethite or other iron oxyhydroxides are routinely removed by chemical reactions: carbonates are dissolved by dilute hydrochloric or acetic acid; iron oxyhydroxides are dissolved by the DCB method (Mehra and Jackson, 1960). However, the separation of clay minerals from fine-grained quartz, which is chemically more resistant than the clay minerals themselves, cannot be performed via dissolution reactions. The only solution to this problem is the physical separation of the phyllosilicate platelets (two-dimensional grains) and the quartz crystals (three-dimensional grains). This behaviour can be expressed in Stokes' formula with the help of the equivalent radius: if the shape is different from the ideal sphere, the equivalent radius is lower than the actual radius of the grain.

DESCRIPTION OF THE NEW PROCEDURE

In high-viscosity liquids platy grains settle down more slowly than three-dimensional grains. This phenomenon can be utilised for the physical separation of phyllosilicate and quartz grains.

The most appropriate, high-viscosity but non-reactive and environmentally friendly liquid was found to be the solution of sodium carboxymethyl cellulose, a jelly material widely used in building industry (aqueous adhesive) as well as pharmaceutics and food industry. The chemical formula of sodium carboxymethyl cellulose is $[C_6H_7O_2(OH)_{2-2.4}(OCH_2COONa)_{1-0.6}]_n$, where n = 100–2000. Solutions of different concentrations were prepared from sodium carboxymethyl cellulose powder, and the most appropriate concentration was found to be the 2% solution.

As separation is enhanced by stirring, centrifugation was applied. Following centrifugation, the jelly material can be easily washed out from the suspended, phyllosilicate-rich material as sodium carboxymethyl cellulose is water-soluble. If it is not completely removed from the sample, it may increase the analysed sodium content of the sample, but this effect can be monitored by analysing the sample both before and after the separation procedure.

After drying the sample, the separation procedure can be repeated as many times as necessary.

AN EXAMPLE: THE ÚRKÚT MANGANESE CARBONATE ORE

The sedimentary Úrkút Manganese Ore Formation is a local facies in the Transdanubian Range zone (Hungary) and is related to the Toarcian anoxic event. Together with the hosting black shale sequence, it was formed in an intrashelf basin of restricted circulation (for a more detailed overview on the geological background of the ore see Weiszburg et al., 2004, and references therein).

The carbonate manganese ore is a mixed clay-sized material, consisting of carbonates (mainly rhodochrosite), a bluish-green 10-Å clay mineral (giving the ore a special bluish-green tint), and other layer silicates, goethite, quartz and apatite in varying proportions. Despite the intensive studies on this green phyllosilicate, reliable chemical data were not available due to separation problems. The ore was selected to be a test material of the new phyllosilicate-quartz separation procedure.

The studied sample originates from the top of the Main Ore Bed, at 283 m of Profile "B_3" of Shaft III (175 m above Baltic sea level), Western Mine Field, Úrkút (for details see Weiszburg et al., 2004).

A three-step separation procedure (Fig. 1) was performed on the ore. Carbonates were removed either by hydrochloric

acid (1 mol/dm^3) or acetic acid (5 wt%). Iron oxyhydroxides were removed by the DCB (dithionite-citrate-bicarbonate) method as described by Mehra and Jackson (1960). In the last step separation of the green clay mineral from quartz was performed by the method described above. Each separation step was documented by a few grams of reference material. X-ray powder diffraction (Fig. 2), infrared spectroscopy (for details see Weiszburg et al., 2004) and full chemical analysis (Table 1) were applied to monitor the course of the separation.

Carbonate removal

The application of either hydrochloric acid (1 mol/dm^3) or acetic acid (5 wt%) treatment resulted in two separation branches (Fig. 1). 2000 ml of 1 N hydrochloric acid was added to 100 g air-dry sample. The solution was first shaken in an ultrasonic cleaner for 2 hours and was centrifuged at 3000 rpm for 2 hours afterwards. The precipitate was thoroughly cleaned by distilled water and then centrifugation was repeated. Finally, the precipitate was dried at 37 °C for twelve hours. As carbonates were not completely removed, the complete procedure was repeated. In the case of the acetic acid treatment, 350 ml of 5% acetic acid was added to 50 g air-dry sample. The solution was kept at room temperature for 60 days. Conditions of centrifugation, cleaning, second centrifugation and drying were the same as for the hydrochloric acid treatment. The duration of the acetic acid treatment was long enough to remove all carbonates from the sample.

Goethite removal

In the second step, goethite and other iron oxyhydroxides were removed by the DCB (dithionite-citrate-bicarbonate) method as descrybed by Mehra and Jackson (1960), with the following proportions of chemicals: to each 10 g of air-dry sample 40 ml 3 M trisodium citrate dihydrate ($Na_3C_6H_5O_7 \times 2H_2O$) solution and 5 ml 1 M sodium bicarbonate ($NaHCO_3$) solution were added. This solution was heated up to 75–80 °C, strictly keeping the temperature below 80 °C in order to prevent the precipitation of iron

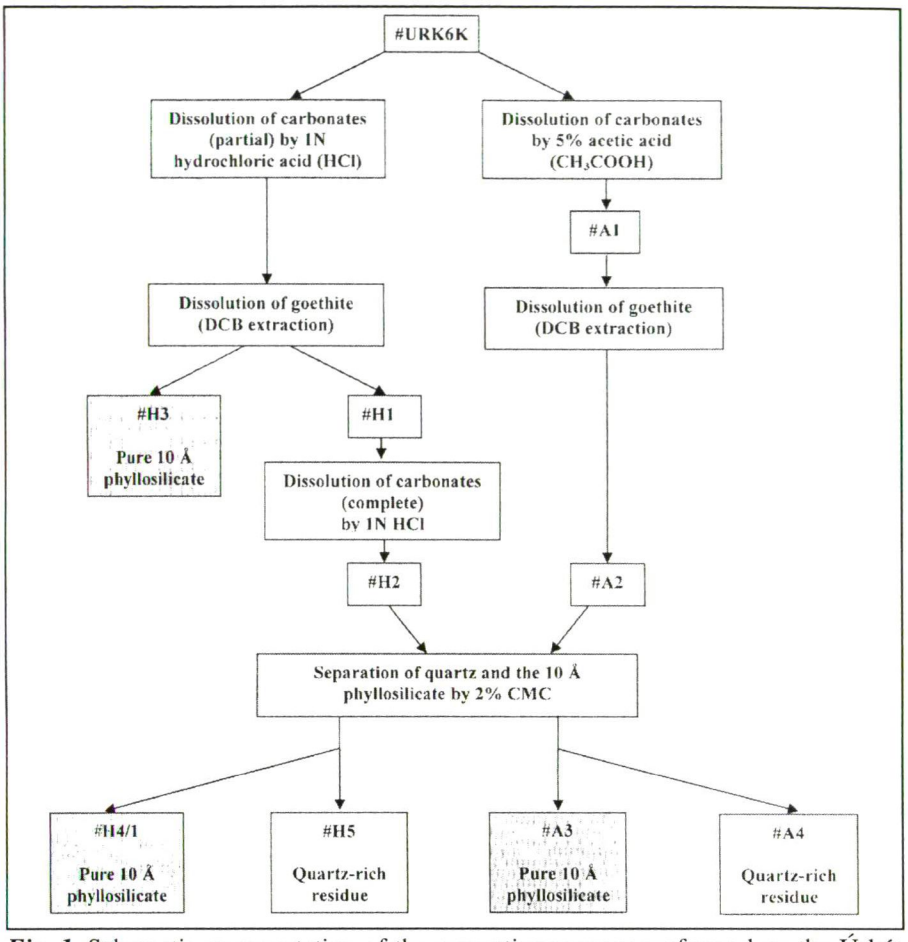

Fig. 1. Schematic representation of the separation process performed on the Úrkút manganese carbonate ore, sample #URK6K (BE81861: powdered reference sample in the Natural History Museum of the Eötvös Loránd University).
The starting material was 100 g in the case of the HCl separation branch and 50 g in the case of the acetic acid separation branch. Shaded boxes represent the pure samples subjected to detailed investigation. (BE81862: reference samples on the hydrochloric separation branch; BE81863: reference samples on the acetic acid separation branch; BE81864: solution remaining after goethite removal on the acetic acid separation branch.)

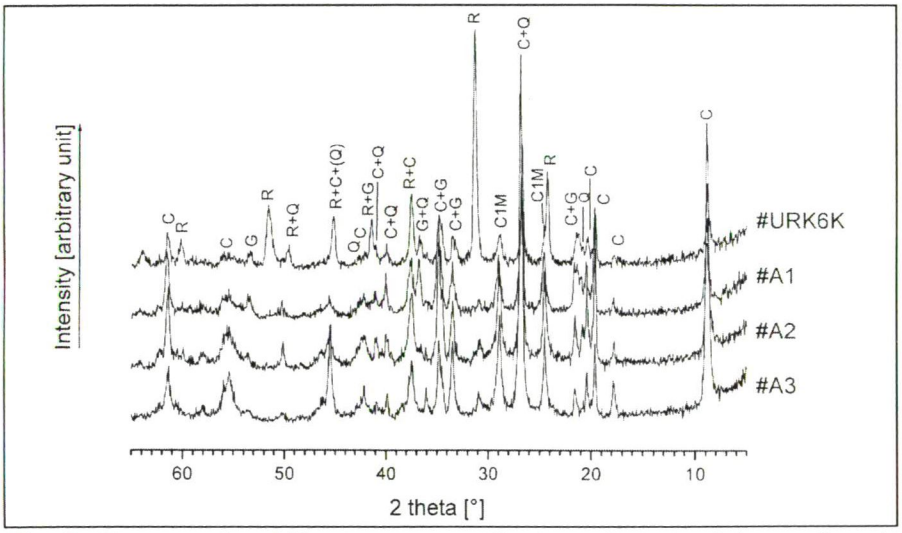

Fig. 2. X-ray powder diffraction pattern (Cu K_α radiation) of the acetic acid separation branch. Note that all separation steps left the mica structure unoffended. C: celadonite, C1M: celadonite reflections characteristic of the 1M polytype, G: goethite, Q: quartz, R: rhodochrosite.

Table 1. Chemical analyses of sample #URK6K and its derivatives. Analysts: Bartha, A. and Bertalan, É. (Geological Institute of Hungary). Data are given in weight percent, except for lithium content that is given in μg/g. Separation steps and the weight of samples analysed are also listed.

Sample	#URK6K	#URK6Kca	#A1	#A1ca	#A2	#A3	#H3	#H2	#H2ca	#H4/1
bulk rock	x	x								
separated from										
carbonates			x	x	x	x	(x)	x	x	x
goethite					x	x	x	x	x	x
quartz						x				x
SiO_2	29.70		45.50		55.20	53.60	52.90	54.80		53.90
TiO_2	0.08		0.14		0.08	0.06	0.07	0.09		0.09
Al_2O_3	1.51		2.52		2.74	2.18[1]	2.53	2.49		2.84
Fe_2O_3 [2]	14.20		25.80		16.20	*15.89*	*18.67*	15.60		*16.20*
FeO [2]	1.39		1.62		2.29	*2.23*	*2.62*	2.25		*2.29*
MnO	21.30	21.26	1.26	1.24	0.84	0.40	0.33	0.35	0.35	0.28
CaO	2.97		0.83		0.86	0.63	0.06	0.06		0.06
MgO	3.41		5.32		6.27	6.44	7.26	5.83		6.46
Na_2O	0.07		0.08		0.13	0.07	0.10	0.05		0.22
K_2O	4.54		7.15		8.23	9.59	9.56	8.28		9.29
LOI	18.30		7.81		6.72	7.31	5.07	8.13		6.30
H_2O-	0.40		0.78		0.47	0.00	0.84	1.20		1.18
P_2O_5	0.79		0.89		0.43	0.23	<0.02	<0.02		<0.02
SO_3	0.30		<0.02		<0.02	0.09	<0.02	0.05		0.01
BaO	0.01		0.01		0.01	0.01	0.01	0.01		0.00
SrO	0.01		0.01		0.01	0.00	0.00	0.00		0.00
Σ	98.98		99.71		100.48	98.73	100.02	99.18		99.12
Fe_2O_3 (total)	15.7	15.68	27.6	27.44	18.7	18.4 [3]	21.6	18.1	18.05	18.7
Li [μg/g]	16	16		21.2				28	28	
Sample weight [g][4]	3.36		2.01		1.79	0.18	0.79	4.04		1.24

Two kinds of sample digestion were applied. Sample names with ending "ca" were digested with concentrated acids. To 0.25 g of sample 1 ml of concentrated HCl, HNO_3 and H_2F_2 each were added to decompose silicate structures and in a second step 10 ml of saturated boric acid was added to the solution to bind excess fluoride. The amount of iron, manganese and lithium was determined in these samples. All other samples were fused with lithium metaborate before the analysis.
[1]: Low value. Relative to the analysis #A2, the amount of Al should have increased as a result of relative enrichment in celadonite / removal of quartz, so this value may be erroneous. [2]:In the case of samples #URK6K, #A1, #A2 and #H2, the amount of FeO has been determined by titration. In the case of samples #A3, #H3 and #H4/1, the total amount of iron was given only in the form of Fe_2O_3. This value was divided according to Fe^{2+} : Fe^{3+} = 14 : 86, a ratio obtained on two independent samples (#A2 and #H2) that contained only the phyllosilicate as the iron-bearing phase (calculated values are shown in italics). [3]: Relative to the analysis #A2, the amount of iron should have increased as a result of relative enrichment in celadonite / removal of quartz, so this value may also be erroneous. In general, analysis #A3 seems to be the least reliable data set, which might be accounted for the small amount of sample analysed. [4]: In those cases where two kinds of digestion were applied, the total amount of sample is given at the lithium metaborate digestion.

sulfides. After adding 1 g of sodium dithionite ($Na_2S_2O_4$) to the solution (for each 10 g of sample), the solution was stirred for 5 minutes. This step was repeated two more times and the final precipitate was free of goethite and free of FeOOH (i.e. containing less than 1%), as confirmed, beside the other monitoring methods, by carbon paste electroactive electrode (CPEE) voltammetry (Tomaš Grygar, personal communication; for the detailed description of the method see Grygar et al., 2002), too.

On the hydrochloric acid separation branch, a part of the sample remained in suspension in the cleaning solution during the DCB extraction. The suspended material turned out to be pure 10-Å phyllosilicate (sample #H3).

Separation of the green clay mineral from quartz

For each gram of sample to be separated, 10 ml 2% solution of the sodium carboxymethyl cellulose was added. The amount of quartz-phyllosilicate mixture subjected to this separation step was 30 g on the hydrochloric acid separation branch and 8 g on the acetic acid branch. The centrifuge was run at 500 rpm for three hours. As a result, the phyllosilicate platelets remained in suspension while the rounded quartz grains settled down. After the separation, the solution was washed out from the suspended fraction and the sample was dried. For greater effectiveness, this separation step was repeated two more times.

Analytical methods

X-ray powder diffraction patterns were obtained at the Department of Mineralogy, Eötvös Loránd University (Budapest). Diffraction patterns were obtained on a SIEMENS D5000 diffractometer of Bragg-Brentano

geometry, equipped with secondary pyrolithic graphite monochromator and applying Cu K_α radiation. Counting time was 10 s per step, step size was 0.05°. For testing the quartz content of the pure samples, precision curves were also recorded, with 10 s detection time and 0.02° step size.

Bulk chemical analyses (inductively coupled plasma atomic emission spectrometry, ICP-AES) of the powdered samples were performed on a Jobin Yvon JY70 spectrometer at the Geological Institute of Hungary (MÁFI) by András Bartha and Éva Bertalan. The amount of FeO was determined by titration.

All samples (except for samples #URK6Kca, #A1ca and #H2ca) were fused with lithium metaborate before the analysis. In the case of samples #URK6Kca, #A1ca and #H2ca concentrated acids (1 ml of cc. HCl, HNO_3 and H_2F_2 each were added to 0.25 g of sample) were added to decompose the silicate structures and in a second step 10 ml of saturated boric acid was added to the solution to bind excess fluoride.

Results

The effectiveness of the last separation step is demonstrated by Fig. 3, in which the X-ray powder diffraction patterns (26.0–27.5° 2θ range, including the most intensive quartz reflection, 3.34 Å/101/100%) of the "phyllosilicate-rich" (sample #H4/1) and "quartz-rich" (sample #H5) phases are displayed. X-ray powder diffraction patterns revealed that samples #H4/1 and #A3 still contain minor quartz while in case of sample #H3, the quartz content is below the detection limit (i.e. less than about 1 volume percent; Fig. 3B).

In order to give a raw quantification on the effectiveness of the separation method, first the initial ratio of quartz and phyllosilicate was calculated using bulk chemical data for the whole rock and the "pure" sample #H4/1. The mineral composition of the studied sample (based on chemical data) is the following: carbonate 41 wt%, celadonite 43 wt%, goethite 8 wt%, quartz 6 wt%, apatite 2 wt% and pyrite 0.23 wt%. (For further details see Weiszburg et al., 2004.) The first two separation steps do not change the phyllosilicate/quartz ratio, thus before

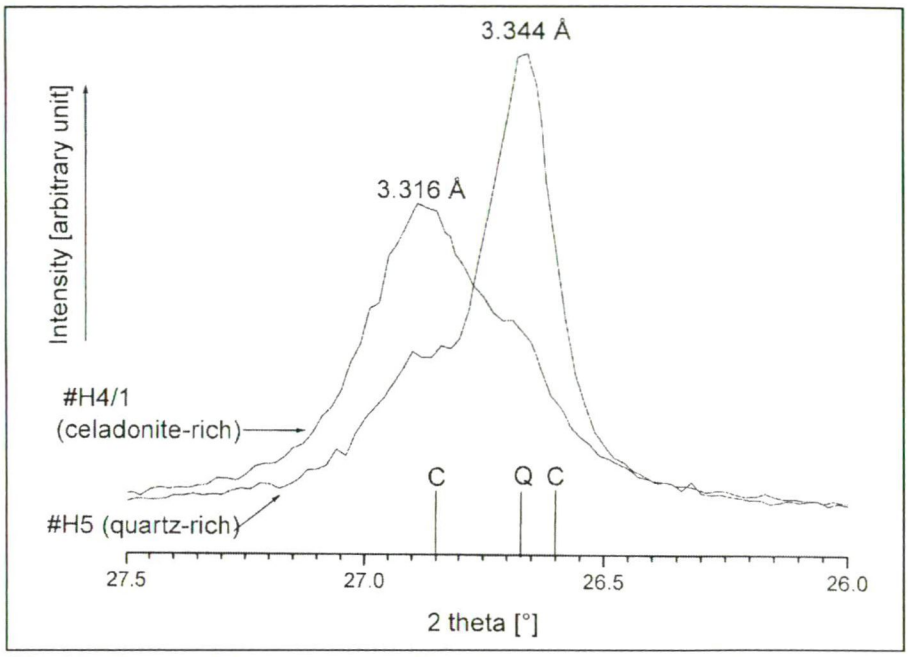

Fig. 3A. Precision XPD patterns (Cu K_α radiation) of the end-products of the last separation step (mica-quartz separation) of the Úrkút green mica (hydrochloric acid separation branch).
Sample #H4/1 was the suspended fraction (third centrifugation) and sample #H5 was the settled fraction (first centrifugation) in the 2% CMC solution. Sample #H4/1 is enriched in celadonite and the most intensive quartz reflection appears only as a shoulder while sample #H5 is strongly enriched in quartz. Pattern #H5 was calibrated to the most intensive quartz reflection (3.342 Å, ICDD no. 33-1161) and pattern #H4/1 was fitted to the other pattern by the undisturbed celadonite peaks.

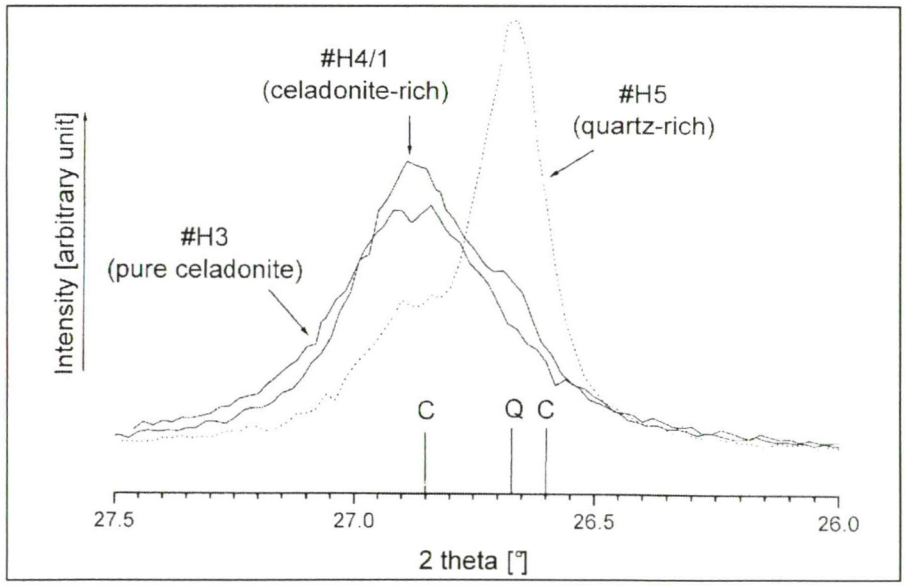

Fig. 3B. Precision XPD patterns (Cu K_α radiation) of the two pure samples (#H3: accidentally separated suspended material during goethite removal, #H4/1: end product of the three-step separation) on the hydrochloric acid separation branch.
The quartz-rich separate (#H5) was used for calibration: it was calibrated by the most intensive quartz reflection (3.342 Å, ICDD no. 33-1161) and the others were fitted to it by the undisturbed celadonite reflections. Compared to the quartz-free sample #H3, the quartz shoulder on the #H4/1 pattern indicates quartz content just above the detection limit of X-ray powder diffraction. Note that the small shoulder on the lower angle side of the celadonite peak of sample #H4/1 does not coincide with the 3.35 Å (26.6° 2θ) peak position given as a celadonite peak e.g. in ICDD no. 17-521.

Table 2. Changes in the calculated formula of the green mica of sample #H4/1 as a function of SiO_2 extracted from the analysed data.

SiO_2 (wt%)	0	1	2	3	4	5	6	7
Composition (pfu)								
K	0.87	0.89	0.90	0.91	0.92	0.94	0.95	0.97
Na	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
Ca	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Fe^{3+}	0.90	0.91	0.92	0.93	0.95	0.96	0.98	0.99
Mg	0.71	0.72	0.73	0.74	0.75	0.76	0.77	0.78
Al	0.22	0.20	0.19	0.17	0.15	0.13	0.11	0.09
Fe^{2+}	0.14	0.14	0.14	0.15	0.15	0.15	0.15	0.16
Mn	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
Ti	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.01
Si	3.97	3.95	3.93	3.91	3.89	3.86	3.84	3.82
Al	0.03	0.05	0.07	0.09	0.11	0.14	0.16	0.18
Fe^{3+}	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
O	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
OH	2.00	2.00	2.00	2.00	2.00	2.00	2.00	2.00
Occupancy (pfu)								
T	4.00	4.00	4.00	4.00	4.00	4.00	4.00	4.00
O	1.99	2.00	2.01	2.01	2.02	2.03	2.03	2.04
IL	0.91	0.92	0.94	0.95	0.96	0.98	0.99	1.00
Charges (pfu)								
T	15.97	15.95	15.93	15.91	15.89	15.86	15.84	15.82
O	5.11	5.12	5.13	5.14	5.14	5.16	5.16	5.17
IL	0.92	0.93	0.94	0.95	0.97	0.98	1.00	1.01
Total	22.00	22.00	22.00	22.00	22.00	22.00	22.00	22.00

T: cations in tetrahedral positions; *O*: cations in octahedral positions *IL*: interlayer cations.
Any apparent discrepancy between the sum of atoms in a given structural site and the occupancy and thus charge values is due to rounding to two decimal digits.

quartz removal the ratio of the two mineral phases was 43:6.

To estimate the increase of the phyllosilicate content during the third separation step, chemistry data of samples #H2 and #H4/1 (having different clay/quartz ratios, as sample #H4/1 was derived from sample #H2 by the third separation step) have been compared. Expressing the increase of the main oxide components invariant to quartz removal (K_2O, MgO, Al_2O_3, Fe_2O_3) as a percentage of the corresponding values in #H2, we can get an estimate on the actual increase in the phyllosilicate content. Reasonable estimates could be obtained from Al_2O_3, MgO and K_2O values. The remaining quartz content is 1.9, 4.7 and 3.5 wt%, calculated from Al_2O_3, MgO and K_2O, respectively. (The three numbers correspond to a decrease of quartz content by 85%, 62% and 72%, respectively.) These values result from three full cycles of separation but, naturally, the separation procedure can be repeated an arbitrary number of times; the more times repeated, the greater effectiveness will be achieved.

Chemical formulae of the phyllosilicate were calculated from the analyses of samples #A3, #H3 and #H4/1. The phosphate contents (P_2O_5 = 0.23–0.89%) of the untreated sample (#URK6K) and the members (#A1, #A2 and #A3) of the acetic acid separation branch indicate the presence of some phosphate-bearing phase, most probably apatite. The amount of CaO was thus reduced according to the P_2O_5 content in the analysis #A3 before the formula calculation. The hydrochloric acid treatment obviously removed the phosphate-bearing phase from the sample as the phosphate contents are below detection limit ($P_2O_5 < 0.02\%$) in samples #H2, #H3 and #H4/1. The minor quartz content of samples #A3 and #H4/1, evident from the X-ray powder diffraction patterns (Fig. 3), where not considered when calculating these formulae. The chemical data and the calculated formulae of the phyllosilicate are published in Tables 3 and 4, respectively, of Weiszburg et al. (2004).

Discussion

Sheet silicates are classified upon their chemical composition (Bailey, 1980, 1986; Rieder et al., 1998) and thus reliable chemical data are vitally important when classifying a mineral. Based upon the formulae calculated from the analyses #A3, #H3 and #H4/1 the bluish-green mica of Úrkút can be classified as celadonite according to the three latest mica nomenclatures (AIPEA – Bailey, 1980, 1986; IMA – Rieder et al., 1998). For a detailed numerical and graphical presentation on the classification of this mica see Weiszburg et al. (2004).

The effect of quartz impurity on the calculated formula was tested in the case of sample #H4/1. Different amounts of SiO_2 (1–8 wt%) were subtracted from the chemical data of sample #H4/1 and the formulae were recalculated (Table 2). The tetrahedral Al substitution, the number of the octahedral and interlayer cations (except octahedral Al) increase, and

consequently, the charge of the tetrahedral cations decreases and that of the octahedral and the interlayer cations increase (Fig. 4). The subtraction of SiO_2 from the analytical data thus leads to an almost diagonal shift (nearly parallel to the tetrahedral charge axis) right upward the *T–O–IL* charge diagram. The higher the supposed quartz content, the higher the number of cations is in the interlayer and the closer the Úrkút celadonite is to an ideal mica. However, these changes are not significant in this case, as the shift does not lead to the mica plotting in the glauconite field (of either the AIPEA nomenclature, Bailey, 1980; or the IMA classification; Rieder et al., 1998).

Although in this case quartz impurities left the chemistry-based classification of the mineral unaffected, in other cases, e.g. for micas with compositions near the glauconite-celadonite boundary (defined by any of the three nomenclatures), a few percent of quartz can lead to misclassification of the mineral.

A possible effect of the separation medium (sodium carboxymethyl cellulose solution) may be the sodium contamination of the sample. In the case of the hydrochloric acid separation branch samples, the Na_2O content slightly increased: from 0.053 wt% in sample #H2 to 0.221 wt% in sample #H4/1. At this order of magnitude (tenth or hundredth wt%) the Na increase due to the enrichment of the layer silicate relative to quartz is insignificant, thus the phenomenon might be assigned to imperfect washing of the sample after the separation. It should be kept in mind, however, that for small quantities the analytical error of the measurement is also higher. In case of the acetic acid separation branch samples the amount of Na_2O decreased from 0.126 wt% in sample #A2 to 0.068 wt% in sample #A3. This phenomenon can only be explained by a large analytical error in sample #A3 (originating from, as already mentioned above, from the small amount of sample analysed). It can be concluded that sodium contamination is negligible in this procedure.

A slight sodium contamination was observed during the goethite removal, too, when trisodium citrate dihydrate, sodium bicarbonate and sodium dithionite were the sodium-bearing chemicals. On the acetic acid separation branch, the Na_2O content increased from 0.076 wt% in sample #A1 (by 66%) to 0.126 wt% in sample #A2, which again cannot be explained by the relative enrichment of the layer silicate due to goethite removal. However, this sodium contamination is considered insignificant from the point of view of the layer silicate, too.

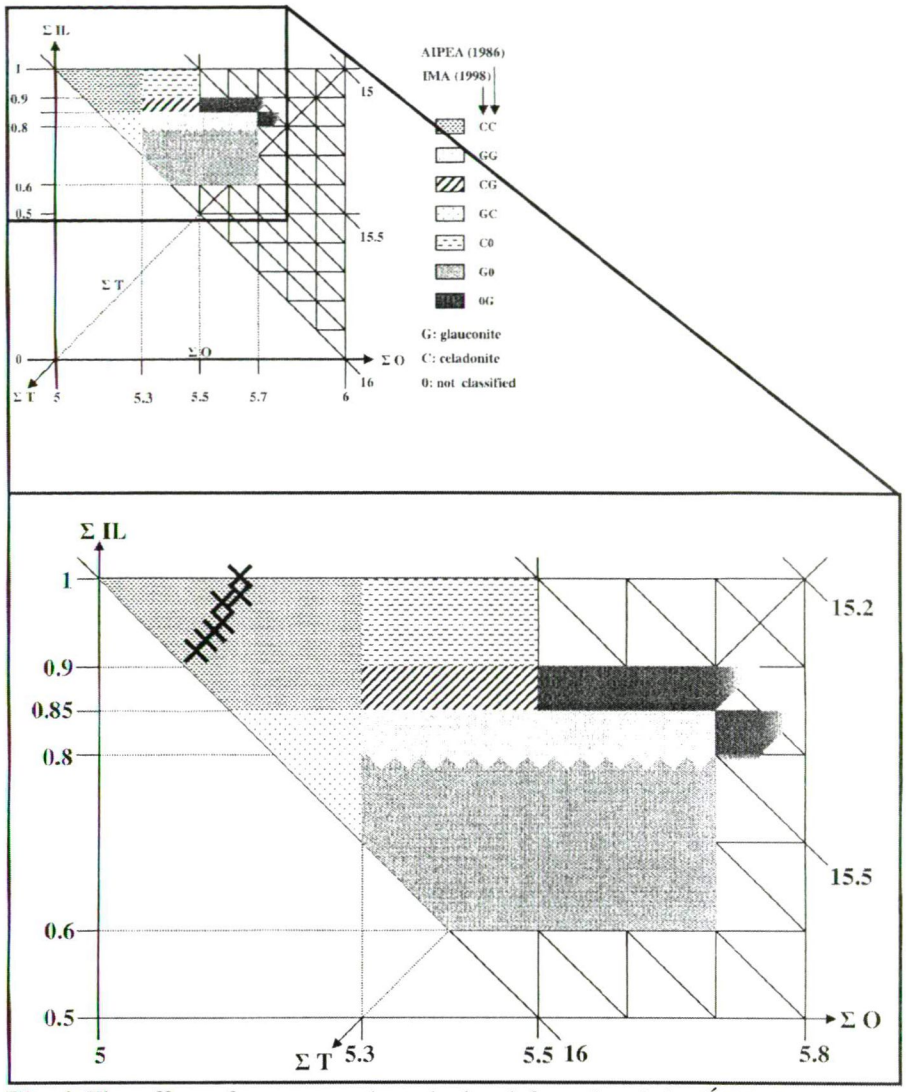

Fig. 4. The effect of quartz on the calculated formula of the Úrkút green mica (sample #H4/1).
The lowermost cross on the left corresponds to the original data (i.e. no quartz contamination assumed), while the uppermost cross on the right corresponds to 6 wt% quartz supposed in the sample. Each cross represents 1 wt% difference in the quartz content.

Conclusions

In this paper, a new procedure for the separation of layer silicates from quartz in clay-sized materials was introduced and tested on the Úrkút manganese carbonate ore. It has been shown that only minor sodium contamination may rise from the application of sodium-bearing chemicals in the case of micas (chemically stable structures in contrast with the extremely sensitive smectites) and that the effects of quartz impurities may be quite apparent in the crystal chemical classification of micas.

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